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“Step Out From the Old to the New”

IS 543 (1968): Cottonseed Oil [FAD 13: Oils and Oilseeds]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS : 543 – 1968
(Superseding IS : 3472 – 1966)
(Reaffirmed ~~2004~~
2009)

Indian Standard
SPECIFICATION FOR
COTTONSEED OIL
(Second Revision)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR COTTONSEED OIL

(*Second Revision*)

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AMENDMENT NO. 4 NOVEMBER 1988

TO

**IS : 543 - 1968 SPECIFICATION FOR
COTTONSEED OIL**

(*Second Revision*)

(*Page 5, clause 2.1.1*) — Substitute the following for the existing clause:

'2.1.1 Refined Cottonseed Oil — Cottonseed oil obtained by expression or solvent extraction of cottonseed oil bearing material, deacidified either with alkali or by physical refining or by miscella refining using foodgrade solvents followed by bleaching with absorbent earth and/or carbon and deodorized with steam.'

(*Page 6, clause 4.3*) — Substitute the following for the existing clause:

'4.3 Admixture with Other Oils — The material shall be free from admixture with other oils.

4.3.1 The material shall be free from non-edible oils when tested in accordance with 9, 10, 11, 12, 14, 15 and 16 of IS : 548 (Part 2)-1976*.'

[*Page 7, Table 1, Sl No. (vii), col 3 and 7*] — Substitute '0.5' for '0.3'.

*Method of sampling and test for oils and fats: Part 2 Purity tests (*third revision*).

**AMENDMENT NO. 5 AUGUST 1995
TO
IS 543 : 1968 SPECIFICATION FOR COTTONSEED
OIL**

(Second Revision)

(*Page 4, Foreword, clause 0.5*) — Add the following clause **0.6** after clause **0.5** and renumber the subsequent clause:

‘0.6 A scheme for labelling environment friendly products to be known as ECO Mark has been introduced at the instance of the Ministry of Environment and Forests (MEF). The ECO Mark shall be administered by the Bureau of Indian Standards (BIS) under the BIS Act, 1986 as per the Resolution No. 71 dated 20 February 1991 as published in the Gazette of the Government of India vide GSR 85(E) dated 21 February 1991. For a product to be eligible for marking with the ECO Mark it shall also carry the Standard Mark of BIS for quality besides meeting additional optional environment friendly (EF) requirements. The EF requirements for cottonseed oil are therefore being included through an amendment.

This amendment is based on the Gazette Notification No. 678 dated 30 August 1994 for Labelling Edible Oils, Tea and Coffee as environment friendly products, published by the Ministry of Environment and Forests.’

(*Page 6, clause 4.4*) — Add the following clauses after **4.4**:

‘4.5 Optional Requirements for ECO Mark

4.5.1 General Requirements

4.5.1.1 The product shall conform to the requirements of quality prescribed under clauses **4.1** to **4.4**.

4.5.1.2 The manufacturers shall produce to BIS environmental consent clearance from the concerned State Pollution Control Board as per the norms laid down under the *Water (Prevention and Control of Pollution) Act, 1974; Air (Prevention and Control of Pollution) Act, 1981; Water (Prevention and Control of Pollution) Cess Act, 1977*, respectively, along with the authorization, if required under the *Environment (Protection) Act, 1986*, while applying for ECO Mark.

4.5.2 Specific Requirements

Amend No. 5 to IS 543 : 1968

4.5.2.1 The product shall not contain aflatoxin, more than 5 mg/kg, when tested by the method prescribed in Appendix B.

4.5.2.2 The pesticide residues, if any, shall not exceed the tolerance limits as prescribed in the *Prevention of Food Adulteration Act, 1954* and *Rules* made thereunder.

4.5.2.3 Only permitted antioxidants not exceeding the quantities specified against each as prescribed under the *Prevention of Food Adulteration Act, 1954* and *Rules* made thereunder, shall be used, if required.

4.5.2.4 The product shall not contain any of the toxic metals in excess of the quantities prescribed in Table 2.

TABLE 2 LIMITS FOR TOXIC METALS

SL NO.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO
i)	Lead, mg/kg, <i>Max</i>	5.0	15 of IS 1699 : 1995*
ii)	Arsenic, mg/kg, <i>Max</i>	0.5	do
iii)	Cadmium, mg/kg, <i>Max</i>	1.0	do
iv)	Mercury (total) mg/kg, <i>Max</i>	0.25	do

* Methods of sampling and test for food colours (*second revision*).

(Page 6, clause 5.1) — Add the following clause 5.1.1 after clause 5.1:

‘5.1.1 For ECO Mark the product shall be packed in such packages which are made from recyclable (that is which can be re-processed to manufacture any useful product) or biodegradable materials.’

(Page 6, clause 6.1) — Add the following clause 6.1.1 after clause 6.1:

‘6.1.1 For ECO Mark, the containers shall be marked with the following information:

- List of identified critical ingredients in descending order of quantity, percent by mass, which shall include ‘made from cottonseed oil’;
- The brief criteria for which the product has been labelled for ECO Mark;
and
- Shelf life of the product.”

(Page 12, clause A-2.5) — Add the following Appendix B after clause A-2.5:

**‘ APPENDIX B
(Clause 4.5.2.1)**

DETERMINATION OF AFLATOXIN

B-1 REAGENTS

B-1.1 Acetone, 70 Percent — 700 ml acetone in 300 ml distilled water.

B-1.2 Acetone, 20 Percent — 200 ml acetone in 800 ml distilled water.

B-1.3 Lead Acetate, 20 Percent — 200 g neutral acetate in distilled water and 3 ml glacial acetic acid, diluted to one litre.

B-2 PROCEDURE

B-2.1 Dissolve 30 g sample in 100 ml hexane.

B-2.2 Extract with 3 × 50 ml 70 percent acetone.

B-2.3 To the extract add 60 ml distilled water and 20 ml lead acetate.

B-2.3 Boil to reduce volume to 150 ml. Cool to about 20°C.

B-2.4 Filter and wash with 20 percent acetone.

B-2.5 Extract filtrate and washings with 3 × 50 ml chloroform.

B-2.6 Pass chloroform layer through anhydrous sodium sulphate.

B-2.7 Concentrate to 50 ml and spot on TLC plate.

B-3 CALCULATION

$$\text{Aflatoxin, mg/kg} = \frac{V \times s \times 1\,000}{v \times m}$$

where

V = volume of extract in ml,

v = volume of extract giving minimum observable fluorescence in μl ,

m = mass of sample in g, and

s = standard toxin giving minimum observable fluorescence in μg .

(FAD 44)

AMENDMENT NO. 6 MARCH 2002
TO
IS 543 : 1968 SPECIFICATION FOR COTTONSEED OIL
(*Second Revision*)

(*Amendment No. 5, page 2, clause 4.5.2.1*) — Substitute '5 µg/kg' for '5 mg/kg'.

(FAD 44)

Indian Standard
SPECIFICATION FOR
COTTONSEED OIL
(*Second Revision*)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 28 August 1968, after the draft finalized by the Oils and Oilseeds Sectional Committee had been approved by the Chemical Division Council and the Agricultural and Food Products Division Council.

0.2 Unlike coconut, groundnut, sesame and mustard oils, raw or unrefined cottonseed oil is not consumed in India for edible purposes. It is used as a salad oil after suitable refining and deodorizing, and is also used in the manufacture of hydrogenated vegetable oil products. Washed cottonseed oil is generally used in the manufacture of soaps. In preparing this standard due consideration has, therefore, been given to the different grades of the oil being used at present for different purposes.

0.3 This standard was originally published in 1954. In its first revision issued in 1966, raw grade oil and an additional grade under washed oil have been introduced. For the washed oil, limits for colour were specified for original as also for bleached oil. The limits of refractive index and iodine value for the oil were revised. Acid value for the refined cottonseed oil was lowered. For raw cottonseed oil, the refining loss was prescribed. As the standard covered only the expressed type of oil and since there was an urgent need for the formulation of an Indian Standard for the solvent-extracted type, IS: 3472-1960* was issued as an emergency standard based on the data made available to the *ad hoc* Consultative Group set up for the purpose. After having decided to normalize the emergency standard, the need for rationalizing the various grades of the oil covered in these standards was keenly felt. This second revision of IS: 543-1954 has been prepared amalgamating its earlier revision and IS: 3472-1960*. As a consequence, IS: 3472-1960* now stands withdrawn.

*Specification for solvent-extracted cottonseed oil.

0.4 In this standard the requirements for the refined and expressed types are based on the extensive laboratory data made available from a collaborative investigation by the laboratories of Hindustan Lever Limited, Bombay; Oil Technological Research Institute, Anantapur; and the Regional Research Laboratory, Hyderabad; covering a large number of representative samples of the cottonseed oil drawn through assistance of the All India Cottonseed Crushers' Association, Bombay. Valuable data were also supplied by the Directorate of Marketing and Inspection, Nagpur.

0.4.1 The method for determination of refining loss was originally adopted from AOCS Official Method Ca 9a-52, Refining loss issued by American Oil Chemists Society, 1960. The method presently prescribed is based on certain simplifications in the apparatus and the procedure recommended by the Regional Research Laboratory, Hyderabad; and the Oil Technological Research Institute, Anantapur. Also, based on their work, limits for the refining loss for the solvent-extracted type oil have now been included.

0.5 The requirements laid down in this standard for the expressed type of the oil correspond to the specifications prescribed by the Directorate of Marketing and Inspection in their Vegetable Oils Grading and Marking Rules, 1955. For the purpose of regulating the quality of the solvent-extracted type of this oil, the concerned Indian Standard on the solvent-extracted type of the oil which is incorporated now had been adopted in the relevant schedule of the Solvent-Extracted Oil, De-oiled Meal and Edible Flour (Control) Order 1967. In so far as the edible grades are concerned, this standard takes care of the provisions of the Prevention of Food Adulteration Rules, 1954, as modified from time to time.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for cottonseed oil for edible and industrial purposes.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given under 2 of IS:548-1964† and also those given below shall apply.

*Rules for rounding off numerical values (*revised*).

†Methods of sampling and test for oils and fats (*revised*).

2.1.1 Refined Cottonseed Oil—Cottonseed oil obtained by the process of expression or solvent extraction which has been refined by neutralization with alkali, bleached with bleaching earth or activated carbon or both, and deodorized with steam, no other chemical agents being used.

2.1.2 Washed Cottonseed Oil—Cottonseed oil obtained by the process of expression which has been neutralized with alkali and subsequently washed and dried.

2.1.3 Semi-refined Cottonseed Oil—Cottonseed oil obtained by the process of solvent extraction which has been neutralized with alkali, and with or without bleaching with bleaching earth or activated carbon or both, no other chemical agents being used.

3. TYPES AND GRADES

3.1 The material shall be of the following types and grades:

a) *Expressed*:

Refined Grade,
Grade 1A (Washed),
Grade 1B (Washed), and
Grade 2 (Raw).

b) *Solvent Extracted*:

Refined Grade,
Semi-refined Grade,
Grade 1 (Raw), and
Grade 2 (Raw).

3.1.1 Refined grades cottonseed oil of both the Expressed and the Solvent-Extracted types are suitable for direct edible consumption.

3.1.2 All the other grades of the Expressed type and the Solvent-Extracted type except Grade 2 (Raw) are suitable for making *VANASPATI* and refined oil and not for direct edible consumption.

3.1.3 Grade 2 (Raw) is intended for industrial uses other than *VANASPATI* and refined oil manufacture.

4. REQUIREMENTS

4.1 Description—The material shall be obtained from good quality cottonseed cake or from clean sound cottonseed kernels from *Gossypium* species, fam. Malvaceae, by a process of solvent extraction or from the cottonseed by a process of expression.

IS: 543-1968

4.1.1 Solvent-extracted oil shall be obtained from the oleaginous material using solvent hexane conforming to IS: 3470-1966*.

4.2 The material shall be clear and free from rancidity, adulterants, sediment, suspended and other foreign matter, separated water and added colouring and flavouring substances.

4.2.1 The clarity of the material shall be judged by the absence of turbidity after keeping the filtered sample at 30°C for 24 hours.

4.3 Admixture with Other Oils—The material shall be free from admixture with other oils, when tested according to the methods prescribed under 20 of IS: 548-1964†.

4.4 The material shall also comply with the requirements given in Table 1.

5. PACKING

5.1 The material shall be supplied in suitable well-closed containers, as agreed to between the purchaser and the supplier.

6. MARKING

6.1 The containers shall be marked with the name, including the type and grade, and weight of the material in the container; manufacturer's name and trade-mark, if any; batch number; and the year of manufacture.

6.2 In addition in the case of the types and grades which are not suitable for direct edible use (*see* 3.1.2 and 3.1.3), the following information shall be suitably marked, either printed on the label affixed to the container or lithographed or stencilled thereon with indelible ink, in a type size of not less than 50 mm:

- a) All grades of the Expressed type and the Solvent-Extracted type except the Refined grades and the Grade 2 (Raw) — 'NOT FOR DIRECT EDIBLE CONSUMPTION';
- b) Grade 2 (Raw) of the Expressed and the Solvent-Extracted type — 'FOR INDUSTRIAL NON-EDIBLE USES ONLY'.

7. SAMPLING

7.1 Representative samples of the material shall be drawn as prescribed under 3 of IS: 548-1964†.

*Specification for hexane, food grade.

†Methods of sampling and test for oils and fats (*revised*).

TABLE 1 REQUIREMENTS FOR COTTONEED OIL
(Clases 4.4 and 8.1)

Sl. No.	CHARACTERISTIC	REQUIREMENT FOR TYPE										METHOD OF TEST, REF TO CL No.
		Expressed					Solvent-Extracted					
		Refined Grade 1A (Wash- ed)	Grade 1B (Wash- ed)	Grade 2 (Raw)	Grade refined 1 (Raw)	Semi-Grade (Raw)	Refined Grade refined 1 (Raw)	Semi-Grade (Raw)	Grade (Raw)			
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)		
i)	Moisture and insoluble impu- rities, percent by weight, <i>Max</i>	0.10	0.10	0.10	0.25	0.10	0.25	0.75	1.5	5 & 6		
ii)	Colour in a 1-in cell on the Lovibond scale, expressed as <i>T</i> + 10 <i>R</i> , not deeper than: a) original oil b) bleached oil	10 —	25 8	35 15	— —	14 —	35 15	— —	— —	13 A-1 of this standard		
iii)	Refractive index at 40°C	—	—	—	1.4630 to 1.4660	—	—	—	—	10		
iv)	Specific gravity at 30°/30°C	—	—	—	0.910 to 0.920	—	—	—	—	11		
v)	Saponification value	—	—	—	190 to 198	—	—	—	—	15		
vi)	Iodine value (Wijs)	—	—	—	98 to 115	—	—	—	—	14		
vii)	Acid value, <i>Max</i>	0.3	0.5	0.5	10.0	0.3	0.75	10.0	25.0	7		
viii)	Unsat. matter, percent by weight, <i>Max</i>	1.5	1.5	1.5	2.0	1.5	1.5	2.0	2.5	8		
ix)	Refining loss, percent by weight, <i>Max</i>	—	—	—	15.0	—	—	15.0	40.0	A-2 of this standard		
x)	Flash point, Pensky-Martens, (closed), °C, <i>Min</i>	—	—	—	—	250	125	100	90	P: 21 of IS: 1448†		

*) Methods of sampling and test for oils and fats (revised).

†) Methods of test for petroleum and its products : Flash point (closed) by Pensky-Martens apparatus.

*Methods of sampling and test for oils and fats (*revised*).

†Methods of test for petroleum and its products: Flash point (closed) by Pensky-Martens apparatus.

8. TESTS

8.1 Tests shall be carried out as prescribed in IS: 548-1964*, IS: 1448† and Appendix A. Reference to the relevant test method is given in col 11 of Table 1.

8.2 Quality of Reagents— Unless specified otherwise, pure chemicals and distilled water (*see* IS: 1070-1960‡) shall be used in tests.

NOTE— 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A P P E N D I X A

[Clause 8.1, and Table 1, Items (ii)(b) and (ix)]

DETERMINATION OF COLOUR AFTER BLEACHING AND REFINING LOSS

A-1. COLOUR AFTER BLEACHING

A-1.0 General— The oil is treated with standard bleaching earth§ under conditions prescribed and colour of the bleached oil determined.

A-1.1 Procedure— Weigh accurately 200.0 ± 0.1 g of oil in a round bottomed three-necked flask. Immerse the flask in a water-bath maintained at 90° to 95°C and slowly bubble carbon dioxide gas through the oil. When the temperature of the oil has reached 90°C, momentarily remove the cork and introduce 2.0 g of the standard bleaching earth§. Start the stirrer and adjust its speed to about 350 rpm. The bottom end of the stirrer should be adjusted to a level at which intimate mixing is ensured. Maintain the temperature at 90°C for 20 minutes and then stop stirring. Remove the flask from the water-bath, allow the oil to cool in an atmosphere of carbon dioxide and filter the oil through filter paper (Whatman No. 1 or equivalent). Determine the colour readings of the bleached oil in $\frac{1}{2}$ -in glass cell, using a Lovibond tintometer as described in 13 of IS: 548-1964*.

*Methods of sampling and test for oils and fats (*revised*).

†Methods of test for petroleum and its products.

‡Specification for water, distilled quality (*revised*).

§Supplied by the Regional Research Laboratory, Hyderabad.

A-2. REFINING LOSS

A-2.0 General—This method determines the loss of free fatty acids, oil and impurities when the sample is treated with alkali solutions under the specific conditions of the test.

A-2.1 Apparatus

A-2.1.1 Balance—torsion type or equivalent, capacity 1 000 g and sensitive to 0.1 g.

A-2.1.2 Refining Cups—1000 ml medium type glass beakers (see IS: 2619-1963*), or seamless stainless steel cups of equivalent size.

A-2.1.3 Refining Apparatus

A-2.1.3.1 The assembly shall consist of flat bottomed metal vessel of about 4 litres capacity to serve as the bath for the refining cups. The bath shall be provided with cup supports so arranged as to hold the cups in a fixed position. The bath shall have suitable hot and cold water connections and adequate heating arrangement so that the change of temperature from cold to hot, as required, may be attained in a minute. Separate baths may also be used for the hot and cold water, if desired.

A-2.1.3.2 The water in the bath shall be kept at a uniform temperature by suitable agitation and the level shall be at least as high as that of the oil and alkali within the refining cups.

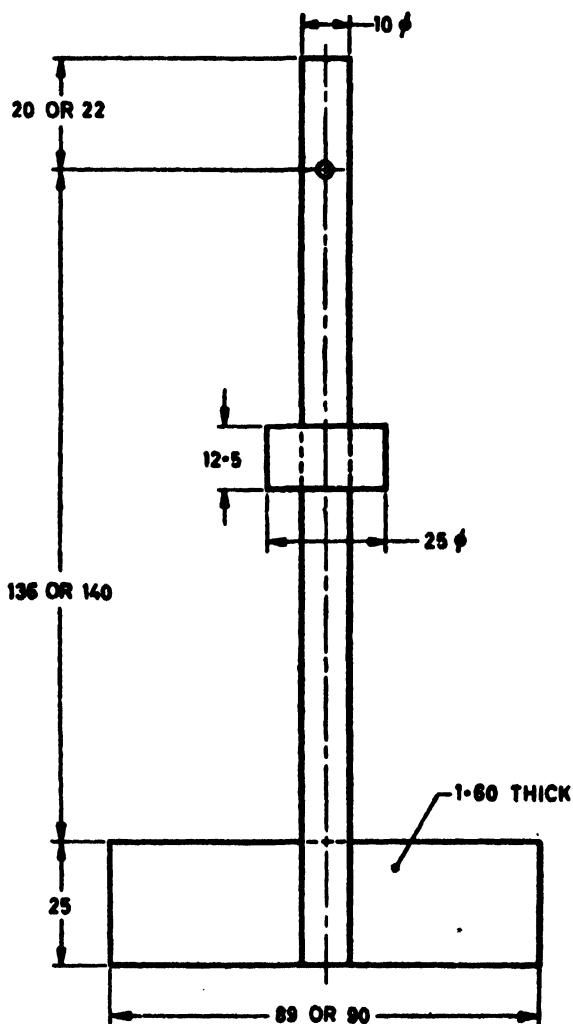
A-2.1.3.3 The paddles run by a suitable electric motor with a variable speed control shall be provided for the agitation of the samples in the refining cups. The paddles for this purpose shall be of stainless steel or nickel-plated copper or brass in accordance with the dimensions shown in Fig. 1. The paddles should be rigid enough so that their bottom is 6 mm above the bottom of the refining cups at all times during the test. Agitation of the samples is required at 70 ± 5 rpm and 250 ± 10 rpm.

NOTE—The slow speed of 70 rpm may also be achieved by hand rotation of the stirrer. The highest speed of 250 rpm may be visually checked by the formation of a conical depression of 4 cm depth extending to about less than half the height of the fluid. Accurate control of the speeds required should, however, be done by placing a white dot of paint on the stirrer shaft and counting the rotation visually against the time with the help of stopwatch.

A-2.2 Reagents

A-2.2.1 Standard Sodium Hydroxide Solution—0.1 N.

*Specification for glass beakers. (Since revised).



All dimensions in millimetres.

FIG. 1 PADDLE FOR REFINING APPARATUS

A-2.2.2 Sodium Hydroxide Solution (20°Bé or about 15 percent, w/w) — This shall be of accurately known sodium hydroxide content, free from carbonate and other impurities, and prepared as follows:

Add 750 g of distilled water to 1 000 g of pure, dry, solid sodium hydroxide. Heat on the steam-bath with occasional stirring for at

least 3 hours. Cool and allow to settle for 24 to 48 hours, keeping the vessel covered to exclude air. During cooling, a portion of the sodium hydroxide which was dissolved in the hot solution crystallizes and precipitates. If the crystals do not separate, the solution was not supersaturated and may not be satisfactory. If properly prepared as described, the solution contains no measurable quantity of carbonate or other impurities. Allow to settle until clear and then carefully decant. Filter through filter paper or asbestos, if the solution is not perfectly clear and protect from the air during filtration to prevent the absorption of carbon dioxide. This solution is then diluted with previously boiled and cooled distilled water to 20°Be (14.36 percent by weight) concentration. The actual strength of the solution shall be determined by weighing and titrating with standard acid. Specific gravity and Baumé test are not sufficiently accurate for the purpose of this test.

A-2.3 Preparation of Sample—The sample container shall be vigorously shaken and the sample thoroughly mixed in order to incorporate and uniformly distribute meal or other sediment. If the oil is cold, heat to 20°C before shaking. Inspect the inside of the can to make sure that no sediment remains clinging to the sides or bottom. If any sediment is found, remove it completely (cut the can open, if necessary) and incorporate thoroughly with the oil. The uniform incorporation and distribution of settlings and suspended matter are very significant in determining the accuracy of the final refining loss.

'CAUTION—SAMPLES SHALL NOT BE ALLOWED TO COME IN CONTACT WITH COPPER SINCE THE COLOUR MAY THEREBY BE AFFECTED'

A-2.4 Procedure

A-2.4.1 Determination of the Acid Value—Determine the acid value as prescribed in 7 of IS: 548-1964*.

A-2.4.2 weigh 500 g of thoroughly mixed sample into the refining cup and place it in the water-bath. Remove any persistent foam in the oil before adjusting the final weight. Fill the water-bath to the specified height with tap water.

A-2.4.3 Start the stirrer and run it at the high speed of 250 rpm and add as quickly as possible the calculated amount of alkali (*see Note*). Continue the high speed agitation for 45 minutes.

NOTE—The amount of sodium hydroxide in grams required for 100 grams of the oil is:

$$\frac{\text{Acid value}}{8.73} \times 0.77$$

*Methods of sampling and test for oils and fats *revised*.

A-2.4.4 Change the agitation to slow speed of 70 rpm and increase the water-bath temperature to 63° to 67°C. This temperature change should be completed in one minute. This may also be done by adding 2.5 litres of boiling water to the outer vessel. Ensure that the level of water in the outer vessel is 5 cm above the oil level inside the cup. Continue stirring and maintain the temperature for 12 minutes.

A-2.4.5 Continue the agitation, remove the stirrer, drain thoroughly against one edge of the stirrer and allow the refined oil in the cup to settle overnight.

A-2.4.6 Weigh the cup and the contents and deduct this weight from the total weight at start to obtain evaporation loss.

A-2.4.7 Decant the refined oil in another tared refining cup and drain the soap stock for 30 minutes by inverting the soap stock cup in the refined oil cup. If the soap stock is too soft to drain, pour off several times. Weigh the soap stock immediately to prevent any moisture loss.

A-2.4.8 Melt the soap stock in water-bath at 75° ± 2°C without stirring for 30 minutes and then cool in cold water (15° to 20°C) for 15 minutes till it is chilled. Decant all possible oil in a tared beaker. Repeat remelting until not more than 1.5 g is recovered. Record the weight of all the oil thus obtained. If oil is difficult to remove due to soft soap stock, use a pipette.

A-2.4.9 Weigh the refined oil and filter through a specified filter into a clean dry container.

A-2.5 Calculation— Calculate the refining loss by the following methods (a) and (b) which should agree within 0.25 percent; and report the average of these results as refining loss, percent:

$$\text{a) Refining loss, percent} = \frac{W_1 - W_2}{5}$$

OR

$$\text{b) Refined loss, percent} = \frac{(A+B) - C}{5}$$

where

W_1 = weight in g of crude oil,

W_2 = weight in g of refined oil,

A = weight of soap stock in g,

B = evaporation loss in g, and

C = weight in g of the 50°Be alkali solution added.

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